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PCT/EP2003/011372  
JC12 Rec'd PCT/PTC 22 APR 2005**USE OF WAX-BASED COMPOUNDS IN TONERS AND  
CORRESPONDING TONERS**

## Description

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Use of wax-based compounds in toners

The present invention relates to the use of wax-containing combinations in toners and also toners in which such combinations are present.

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In modern copying processes, it is usual to use toners comprising resins, pigments, charge control agents and waxes and also, if appropriate, flow aids. During copying, the pulverulent toners usually firstly reproduce the image to be copied on a transfer roller, are transferred from there to the copying paper and are subsequently thermally fixed.

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The waxes present in the toner as formulation components act as release agents and anti-offset agents to aid detachment of the toner from the fixing roller, act as adhesion promoters on transfer to the paper and in the production of the toner act as dispersants and thus contribute to homogeneous distribution of the pigments.

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The demand for ever faster copiers requires correspondingly fast-responding toner systems and places high demands on the individual components of the toner formulation.

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The wax components used in toners have hitherto been predominantly hydrocarbon waxes such as polyethylene or polypropylene waxes. These waxes are not able to meet all the demands placed on them by modern fast-running copying machines. In particular, there is a need for wax components having an improved anti-offset action, improved performance in respect of adhesion of the print to the paper and further optimized pigment-dispersing properties.

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JP-A-1 142 560 describes the use of erucamide in liquid toners. Owing to its low hardness, erucamide has only limited suitability for use in toners.

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There has hitherto also been a shortage in the prior art of products having

satisfactory release properties in toner resins without at the same time impairing the optical properties of the resin.

5 It is therefore an object of the present invention to provide a wax-containing combination which avoids the abovementioned disadvantages and can be used to give very good results in toners.

10 The invention therefore provides for the use of a combination of erucamide and one or more wax(es) in toners.

The wax is preferably selected from the group consisting of natural waxes and partially synthetic waxes.

15 The wax preferably has a needle penetration of not more than  $10 \text{ mm}^{-1}$ .

The wax preferably has a melting point of from  $50$  to  $160^\circ\text{C}$ .

20 The invention likewise provides toners comprising a combination of erucamide and one or more waxes.

The wax is in this case preferably selected from the group consisting of natural waxes and partially synthetic waxes.

25 The wax preferably has a needle penetration of not more than  $10 \text{ mm}^{-1}$ .

The wax preferably has a melting point of from  $50$  to  $160^\circ\text{C}$ .

30 For the present purposes, the term "wax" is used in accordance with a definition of the Deutschen Gesellschaft für Fettwissenschaft as a collective term for a series of materials of natural or synthetic (partially or fully synthetic) origin which have the following properties (Fette Seifen Anstrichmittel 76, page 135, 1974):

35 kneadable at  $20^\circ\text{C}$ ,  
solid to hard and brittle,  
coarsely to finely crystalline,  
translucent to opaque, but not vitreous,  
melts above  $40^\circ\text{C}$  without decomposition,  
relatively low viscosity even just above the melting point,

highly temperature-dependent consistency and solubility,  
polishable under light pressure.

Possible natural waxes are, for example, plant waxes such as carnauba or  
5 candelilla wax or waxes of animal origin, e.g. shellac wax. Suitable partially  
synthetic waxes are, for example, bleached montan waxes which may have  
been chemically modified, e.g. by esterification and/or by partial  
saponification. Such products are described, for example, in Ullmann's  
Encyclopedia of Industrial Chemistry, 5th edition, Vol. A 28, Weinheim  
10 1996, in chapters 2.2, 2.3 and 3.1-3.5.

It is also possible to use polar or nonpolar fully synthetic waxes, e.g.  
polyolefin waxes. Nonpolar polyolefin waxes can be prepared by thermal  
degradation of branched or unbranched polyolefins or by direct  
15 polymerization of olefins. Possible polymerization processes are, for  
example, free-radical processes in which the olefins, generally ethylene,  
are reacted at high pressures and temperatures to form more or less  
branched waxes; also processes in which ethylene and/or higher 1-olefins  
are polymerized with the aid of organometallic catalysts, for example  
20 Ziegler-Natta or metallocene catalysts, to form unbranched or branched  
waxes. Methods of these types for preparing olefin homopolymer and  
copolymer waxes are described, for example, in Ullmann's Encyclopedia of  
Industrial Chemistry, 5th edition, Vol. A 28, Weinheim 1996, in chapter  
6.1.1./6.1.2. (high-pressure polymerization), chapter 6.1.2. (Ziegler-Natta  
25 polymerization, polymerization using metallocene catalysts) and chapter  
6.1.4. (thermal degradation).

Polar polyolefin waxes are formed by appropriate modification of nonpolar  
waxes, e.g. by oxidation with air or by grafting-on of polar olefin monomers,  
for example  $\alpha,\beta$ -unsaturated carboxylic acids and/or their derivatives, e.g.  
30 acrylic acid or maleic anhydride. Polar polyolefin waxes can also be  
prepared by copolymerization of ethylene with polar comonomers, for  
example vinyl acetate or acrylic acid; also by oxidative degradation of  
relatively high molecular weight ethylene homopolymers and copolymers  
which are not wax-like. Appropriate examples may be found, for example,  
35 in Ullmann's Encyclopedia of Industrial Chemistry, 5th edition, Volume  
A 28, Weinheim 1996, chapter 6.1.5.

Preference is given to the use of a combination of erucamide and waxes

selected from the group consisting of natural and partially synthetic waxes.

The waxes used according to the invention have needle penetration values of not more than  $10 \text{ mm}^{-1}$ , preferably not more than  $5 \text{ mm}^{-1}$ , and dropping points of from 50 to  $160^{\circ}\text{C}$ , preferably from 60 to  $120^{\circ}\text{C}$ , particularly preferably from 70 to  $90^{\circ}\text{C}$ .

Base components present in toners are generally resins based on polyesters or styrene-acrylate copolymers. As charge control agents which aid the transfer of the toner from the photo roller to the paper substrate, use is made of, for example, quaternary ammonium salts for a positive charge and, for example, aluminum-azo complexes for a negative charge of the toner powder. To aid powder flow, small amounts of finely divided silicas can be added to the toner powder.

Depending on the desired color, suitable black or color pigments are added to the toners at the stage of the thermoplastic mixture.

#### Examples

The needle penetration values reported below were determined in accordance with DIN 51579, and the dropping points were determined in accordance with DIN 51801/2.

Examples 1 to 3:

90 parts by weight of a styrene-acrylate resin (grade CPR 100, manufacturer: Mitsui; glass transition temperature:  $60^{\circ}\text{C}$ ; MFR/ $140^{\circ}\text{C}$ : 5 g/10 min) were homogeneously mixed with 4 parts by weight of a black pigment (carbon black having a mean particle size of  $2 \mu\text{m}$ ; manufacturer: Timcal), 1 part by weight of a charge control agent (<sup>®</sup>Copy Charge N4S, manufacturer: Clariant GmbH) and 4 parts by weight of an erucamide/wax combination used according to the invention as shown in Table 1 in a kneader. This mixture was then comminuted to give a toner powder having a mean particle size of  $12 \mu\text{m}$  ( $100\% < 20 \mu\text{m}$ ). 0.5 part by weight of a silica-based flow aid (grade HDK, manufacturer: Wacker) was then added to the toner powder. 5 g of this now free-flowing mixture were mixed with 95 g of iron powder and introduced into the reservoir of a copier.

Toner powder was then applied to a sheet of paper over an area of 20 × 100 mm by means of the photomagnetic roller in the copier. This image was then fixed by means of a roller arrangement comprising a rigid heatable roller and an elastic cold roller at 160°C and a linear velocity of 150 mm/s. A further white sheet was subsequently passed through the hot pair of rollers and examined for toner residues. No "ghost pictures" were observed on the white sheet.

Table 1: Erucamide combinations used

Example	Wax	Weight ratio of erucamide:wax
1	®Licowax E (Clariant GmbH) (NP: about 1, dropping point: about 82°C)	10:1
2	®Licowax F (Clariant GmbH) (NP: about 1, dropping points about 79°C)	10:1
3	Carnauba wax (NP: about 1, dropping point: 83°C)	10:1